ELECTROLESS Ni-P PLATING WITH CONTINUOUSLY CONTROLLED CONSTITUENTS

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Electroless plating bath consists of the metal salt to be plated, reducing agent and complexing agents. A Ni-P plating bath contains nickel sulfate, sodium hypophosphite and organic salts, such as sodium lactate, malate, etc. In the bath, Ni-P deposit is obtained by the oxidation-reduction process of Ni^{2+}, $H_2PO_2^-$ and H_2O, whereas $H_2PO_3^-$ and H_2 gas are generated as byproducts. Also, the solution pH changes. The organic constituents, which may affect the physical properties of deposited alloy, are not consumed theoretically.
In fact, the Ni-P deposited layer is affected greatly by changing the phosphite concentration on the time of operation. In other words, the Ni-P deposit having adequate properties on chemical composition, especially the phosphite concentration is controlled at certain levels. Thus, a method for the precise concentration control of constituents was studied.

EXPERIMENTAL

A plating tank of 50 liter in volume, connected with a solution regeneration system, named "Auto-Drain" (1) was conducted. Compositions of the plating bath and three replenishments are listed in **Table 1**. Of these, replenishing solution (I) was prepared by a "regenerated solution" which was reported previously (2).

RESULTS AND DISCUSSION

The plating rate is decreased and the physicochemical properties of deposit are varied when the phosphite concentration in the bath increases with time, and hence the plating solution is replaced at certain intervals.

The "Auto-Drain" system is being operated at shops of under layer plating of hard-discs, to keep the solution compositions (3). Therefore, its system combined with a regeneration process (2) was employed for experiments.

Figure 1 shows the results by using the combined system. The abscissa shows the nickel deposited from a liter of the plating solution in mol. It is clear that the deposition rate and the P content in the deposit are almost unchanged during the operation. The solution of 0.1655 liter in volume was discharged and the replenishments of 0.2995 liter in volume was charged at an interval of the nickel consumption of 0.053 mol or 3.1 grams. It was repeated 3503 times during experiment, counted for 580 liters of discharge and 1049 liters of replenishments. Water of 432 liters was vaporized, and hence deionized water of 37 liters in volume was charged.

Figure 2 shows the time variation of the concentration of some constituents in the plating bath. The results indicate that the solution composition, and hence the chemical composition of deposit could be kept constant by the process, resulting in continuous operation for many hours with no detriment of the Ni-P deposit. A predictable film property can be expected for the electroless Ni-P plating as same as an electroplating.

Table 1. Compositions of plating bath and replenishments in mols/liter.

	Plating bath	Replenishments I*	II	III
Nickel	0.085	0.317	---	---
Hypophosphite	0.255	0.176	5.283	---
Phosphite	1.140	0.096	---	---
Complexing agents	1.465	1.465	---	---
NaOH	(pH=4.8)	(pH=4.8)	---	4.000

*Prepared by a "regenerated solution" (2).

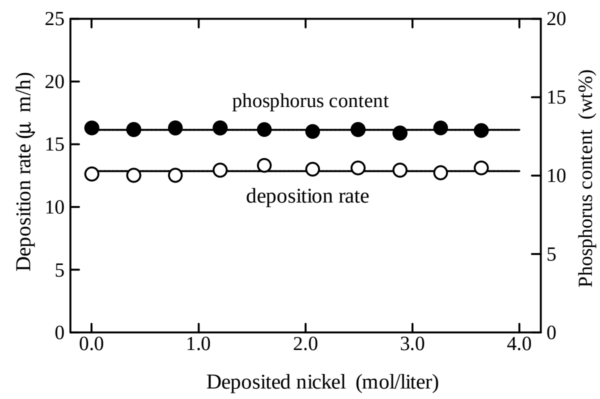

Fig.1 Deposition rate and P content as functions of deposited nickel during operation.

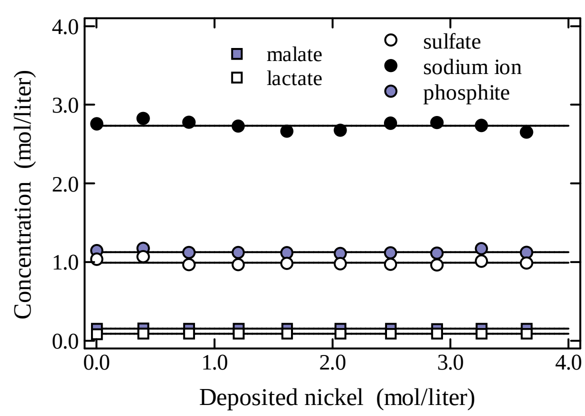

Fig. 2 Concentration of some constituents in the plating bath.

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